

## PATENT COOPERATION TREATY

PCT

## NOTIFICATION OF ELECTION

(PCT Rule 61.2)

From the INTERNATIONAL BUREAU

To:

Commissioner  
 US Department of Commerce  
 United States Patent and Trademark  
 Office, PCT  
 2011 South Clark Place Room  
 CP2/5C24  
 Arlington, VA 22202  
 ETATS-UNIS D'AMERIQUE  
 in its capacity as elected Office

<b>Date of mailing</b> (day/month/year) 30 January 2001 (30.01.01)	
<b>International application No.</b> PCT/CA00/00565	<b>Applicant's or agent's file reference</b> 30177-0007
<b>International filing date</b> (day/month/year) 08 May 2000 (08.05.00)	<b>Priority date</b> (day/month/year) 07 May 1999 (07.05.99)
<b>Applicant</b> MACQUARRIE, Reg et al	

1. The designated Office is hereby notified of its election made:

☒ in the demand filed with the International Preliminary Examining Authority on:  
 06 December 2000 (06.12.00)

☐ in a notice effecting later election filed with the International Bureau on:

2. The election ☒ was  
☐ was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland Facsimile No.: (41-22) 740.14.35	Authorized officer R. E. Stoffel Telephone No.: (41-22) 338.83.38
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## INTERNATIONAL SEARCH REPORT

International Application No

PCT/CA 00/00565

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 A22C13/00

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 A22C C08J C08L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

PAJ, WPI Data, EPO-Internal

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	EP 0 400 484 A (MITSUBISHI RAYON CO., LTD.) 5 December 1990 (1990-12-05) cited in the application page 3, line 51 -page 4, line 29 page 4, line 40 -page 5, line 10; claims 1-20	1-8
Y	EP 0 328 317 A (TAKEDA CHEMICAL INDUSTRIES, LTD.) 16 August 1989 (1989-08-16) page 2, line 19 - line 56 page 3, line 47 - line 55; claims 1-10	1-8

-/--

☒ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

## \* Special categories of cited documents:

- \*A\* document defining the general state of the art which is not considered to be of particular relevance
- \*E\* earlier document but published on or after the international filing date
- \*L\* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- \*O\* document referring to an oral disclosure, use, exhibition or other means
- \*P\* document published prior to the international filing date but later than the priority date claimed

\*T\* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

\*X\* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

\*Y\* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

\*&\* document member of the same patent family

Date of the actual completion of the international search

5 September 2000

Date of mailing of the international search report

15/09/2000

Name and mailing address of the ISA

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Fax: (+31-70) 340-3016

Authorized officer

Permentier, W

## INTERNATIONAL SEARCH REPORT

International Application No

PCT/CA 00/00565

## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No
Y	<p>✓ DATABASE WPI Section Ch. Week 9202 Derwent Publications Ltd., London, GB; Class A11, AN 1985-113524 XP002146576 &amp; JP 60 055039 A (MITSUBISHI ACETATE CO., LTD.), 29 March 1985 (1985-03-29) abstract</p>	1-8
Y	<p>✓ GB 2 000 424 A (UCC) 10 January 1979 (1979-01-10) page 4, line 83 - line 94 page 4, line 124 -page 5, line 29; claims 1-28</p>	1-8
A	<p>✓ EP 0 709 030 A (HOECHST AG) 1 May 1996 (1996-05-01) column 2, line 8 -column 3, line 59; claims 1-10</p>	1,4
A	<p>✓ DATABASE WPI Week 9218 Derwent Publications Ltd., London, GB; AN 1992-147687 XP002146577 &amp; JP 04 089841 A (MITSUBISHI RAYON CO., LTD.), 24 March 1992 (1992-03-24) abstract</p>	1
A	<p>✓ EP 0 795 270 A (VISKASE CORPORATION) 17 September 1997 (1997-09-17) claims 1-20</p>	1
A	<p>✓ DE 12 13 211 B (WOLFF &amp; CO. AG) 24 March 1966 (1966-03-24) the whole document</p>	1,7

# INTERNATIONAL SEARCH REPORT

Int. Patent Application No.

PCT/CA 00/00565

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- "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
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Permentier W

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# INTERNATIONAL SEARCH REPORT

Information on patent family members

In International Application No

PCT/CA 00/00565

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
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			FR 2395912 A	26-01-1979
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			SE 7807276 A	29-12-1978
			ZA 7803674 A	25-07-1979
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			AT 183617 T	15-09-1999
			DE 59506682 D	30-09-1999
			ES 2137433 T	16-12-1999
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			US 5928737 A	27-07-1999
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			CA 2105604 A	14-09-1997

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/CA 00/00565

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP 795270	A	CZ 9700745 A ZA 9702011 A	17-09-1997 30-07-1998
DE 1213211	B	NONE	

## PATENT COOPERATION TREATY

## PCT

## INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference <b>30177-0007</b>	<b>FOR FURTHER ACTION</b> see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.	
International application No. <b>PCT/CA 00/ 00565</b>	International filing date ( <i>day, month, year</i> ) <b>08/05/2000</b>	(Earliest) Priority Date ( <i>day, month, year</i> ) <b>07/05/1999</b>
Applicant <b>MACQUARRIE, Reg et al.</b>		

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 3 sheets.

☒ It is also accompanied by a copy of each prior art document cited in this report.

## 1. Basis of the report

- a. With regard to the **language**, the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.

☐ the international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).

- b. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international search was carried out on the basis of the sequence listing:

☐ contained in the international application in written form.

☐ filed together with the international application in computer readable form.

☐ furnished subsequently to this Authority in written form.

☐ furnished subsequently to this Authority in computer readable form.

☐ the statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.

☐ the statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

2. ☐ **Certain claims were found unsearchable** (See Box I).

3. ☐ **Unity of invention is lacking** (see Box II).

4. With regard to the **title**,

☒ the text is approved as submitted by the applicant.

☐ the text has been established by this Authority to read as follows:

5. With regard to the **abstract**,

☒ the text is approved as submitted by the applicant.

☐ the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.

6. The figure of the **drawings** to be published with the abstract is Figure No.

☐ as suggested by the applicant.

☐ because the applicant failed to suggest a figure.

☐ because this figure better characterizes the invention.

☐ None of the figures.



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Information on patent family members

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JP 4089841	A	24-03-1992	NONE	
EP 795270	A	17-09-1997	US 5695800 A	09-12-1997
			CA 2195694 A	14-09-1997

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Information on patent family members

International Application No

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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP 795270      A		CZ    9700745 A ZA    9702011 A	17-09-1997 30-07-1998
DE 1213211    B		NONE	

EDIBLE CASING FILM FORMULATIONBackground of the Invention

For a number of years, edible films have been used to replace membranes and casings made of animal viscera, in the production of smoked meat products such as ham and sausages. Currently available edible films include collagen films, modified cellulose films and carrageenan-based films.

Collagen films are composed of edible collagen, plasticized with glycerol or other polyols. They are manufactured by extrusion onto a belt followed by drying to remove the moisture. The collagen slurry is applied to the belt cold, and requires high-temperature drying to remove all the excess moisture and bring the film to a final moisture content of approximately 10%. Apart from the difficulty in processing films of this kind, these materials can bring about allergic reactions in certain consumers. Too, being derived from the coum layer of beef or pork, collagen may be an unacceptable casing or film material for religious and dietary reasons.

Modified cellulose films are for the most part composed of modified hydroxypropyl methylcellulose. To date, almost all films of this kind which have been produced are cold water-soluble films which begin to disintegrate on contact with water. For this reason, they have not been used in meat processing.

Carrageenan is a polyanionic polysaccharide derived from red algae. The material is an excellent film-former and has been used in the formulation of edible food packaging films (U.S. Patent No. 4,851,394) and heat-sealable edible films (U.S. Patent No. 5,089,307 - Ninomiya et al.). Films composed primarily of carrageenan additionally exhibit excellent strength, are not soluble in cold water and bind well to meat

However, because carrageenan forms a thermoreversible gel, films composed of a predominant carrageenan proportion tend to disintegrate in hot water, i.e. above 70°C. The hot carrageenan aqueous solution  
5 then re-gels upon contact with cooler water, less than 50°C.

It was one of our objectives to develop carrageenan-based films which can effectively and economically replace edible collagen films currently utilized in ham  
10 and sausage production, while providing high yields and high-quality product.

Collagen film is typically wrapped around hams during production, for ease in netting removal after the ham has been cooked. Collagen films appear to the  
15 consumer to be part of the ham after cooking and, indeed, are usually not taken note of by the average consumer. Many styles of meats are processed using collagen films, including various ham, chicken and turkey products.

#### General Description and Objects of the Invention

20 It is an object of the present invention to provide polysaccharide films which would function well by satisfying all of the following key attributes:

- composed of food ingredients which meet Food Chemical Codex Specifications and are approved for  
25 usage, i.e. GRAS rated in the United States (generally regarded as safe).
- a film that would maintain its composition during the cooking cycle, until the ham forms its own natural protein skin.
- 30 - after cooking, film adheres to the finished product, in that it does not fully disintegrate after a prolonged exposure to hot water or steam.
- film is tender to eat following processing of the meat product.

- film binds to the meat surface during and subsequent to processing.
- film is permeable to flavouring smoke.
- film imparts an appealing shine and colour to the cooked product.

We have found that excellent properties for edible films can be achieved by including, along with the carrageenan, suitable non-thermoreversible gel-forming polymers, preferably Konjac and Gellan gums. Konjac is extracted from the konjac tuber. This polysaccharide hydrates immediately and can form either thermoreversible or non-thermoreversible gels, depending on the conditions under which the gel is formed. Gellan gum is a polysaccharide gum produced from bacterial fermentation. It is a strong gel former and produces gels that are non-thermoreversible in the presence of  $\text{Ca}^{+2}$  and other multivalent anions. Both Konjac and Gellan gums are edible and GRAS rated. We observed that Konjac tends to form gels having a higher degree of elasticity than Gellan, which forms gels having a more brittle texture.

According to present invention, edible films and casing materials comprise a polymer base of carrageenan/Konjac/Gellan, and also a suitable starch, alginate or other polysaccharide, depending on the specific attributes required.

Starch functions to reduce water absorption of the film and it is preferred that a high-amylose starch be used, as high-amylose starch is inherently a better film-former than other starches.

"Alginate" herein refers to the alginates which are commonly used in the food industry, namely polypropylene glycol alginate, sodium alginate or calcium alginate. Alginate in compositions according to the present

invention tend to produce strong films exhibiting good adhesion.

Specific methods and formulations are described in more detail below, but generally we have found the following compositions to provide useful edible films:

	Component	Suggested Range	
		Low (wt.%)	High (wt.%)
	Carrageenan	2	35
	Gellan	0	35
10	Konjac	0	35
	Locust bean gum (LBG)	0	10
	Protein	0	15
	Starch	0	20
	Alginate	0	35
15	Sodium Citrate	1	5
	Sorbitan Monostearate	0.25	3
	Glycerol	1.0	35
	Other Plasticizer*	0	25
	Water	8	35
20	** <u>total</u> of Gellan & Konjac gums should be no less than about 5 wt% and up to about 35 wt%		
	* <u>total</u> of starch + alginate should be no less than about 5 wt% and up to about 35 wt%		
	' polyhydric alcohols, other than glycerol		

25 The synergistic effects of the main polymers, Gellan Konjac and carrageenan, function to produce a film of high tensile strength, hot and cold water resistance and gum substrate adherence.

30 The use of a protein source is desirable to provide the film with both improved adhesion and shine functionality. The presence of some (at least about 5%) of alginatge or starch component is necessary to give sufficient film casing adhesion.

35 Other polysaccharides, as discussed below, may be added to impart specific desired properties to the film. Glyerol was added for its elasticizing effects.

Optionally, calcium chloride or potassium carbonate may be utilized to improve the film strength and hot water resistance.

From other formulations tested it is possible to  
5 significantly alter the relative amounts of each gel-forming polymer in the film to produce films exhibiting some varying degree of hot water solubility.

#### Detailed Description of the Invention

Polymer solutions were prepared for film casting as  
10 follows: glycerol or other plasticizers were added to the requisite amount of hot water. While maintaining the solution at an elevated temperature (80-90°C), then the dry ingredients were added and the solution vigorously  
15 mixed to homogeneity. The polymer solution was then continuously mixed under slow agitation for 1-2 hours to remove trapped air bubbles.

Once the polymer solution appeared to be smooth and free of lumps, it was cast into film, using either a casting box or with a standard draw down bar. Care must  
20 be taken to ensure that the hot solution is cast in a uniform thin layer. In all of the experimental examples given below, film was formed by drawing the solution down onto a hot (90°C) stainless steel belt with a Gardener dye. The films were either air dried overnight at room  
25 temperature, or dried over a hot water bath and then removed for testing.

Films produced this way would initially be tested for thickness using a micrometer (Gauge) and tensile/tear strength.

30 The methods for measuring tear strength and elongation are those specified in ASTM D638. Normally 5

specimens are tested from each of 5 samples with the following conditions

- 20lbf load cell
- Crosshead speed: 2 in./minute
- 5 • Die "C" cut specimens

Films that met these requirements would then be tested for hot water solubility by placing strips of the film in boiling water and then allowing the water to cool with the film sample in it. After cooling, the water  
10 would then be drained off and non-solubilized material would be placed with the drain dish in the drying oven. If the film was reconstituted after drying then the film was deemed to have maintained its structure during exposure to the hot water. If the film was not resistant  
15 to the water then it would become soluble and could be poured off. (Hence no film left after drying)

After a particular sample had been characterized as above, it would be subjected to the same process conditions under which commercial meats are produced. In  
20 particular the film would be wrapped around a particular meat substrate (turkey, ham, chicken) and then the wrapped meat sample dried in a convection oven until a protein skin could be seen forming on the sample. After this the sample was transferred into a steam cooker for  
25 the completion of cooking. The internal temperature of the meat sample would have to reach 70-80°C. After the completion of cooking, the sample was allowed to cool and the final product examined and results recorded. Samples were then packed away under vacuum for shelf-life  
30 testing.

Films according to the invention which we tested on meat film wrap were also produced using the following polysaccharides, supplementary to starch and/or alginate, with Gellan and/or Konjac: agar, modified starches, guar



gum, cellulose gum, modified cellulose gum, gum arabic, pullian and xanthate.

In some of the formulations, the following proteins were included: gelatin, soya protein concentrate, soya protein isolate, whey protein concentrate, albumin, miscellaneous vegetable proteins, collagen and collagen hydrolysates.

The following general observations arose from our experiments:

- 10 (i) Either Gellan and/or Konjac is required for synergistic reaction with the carrageenan and other gums or polysaccharide to provide the desired hot water resistance. The proportion of Gellan and/or Konjac is that amount necessary to  
15 modify carrageenan sufficiently to ensure that the film will not disintegrate upon exposure to hot water. Films could be made out of Konjac or Gellan alone, but these would be far less economical than the combination with other  
20 polysaccharide and in any event would have inferior physical properties, e.g. poor film strength, susceptibility to hydration, etc.
- (ii) The presence of starch or alginate is essential in these compositions to impart proper adhesion at  
25 the final film in its intended use as food film and casing.
- (iii) Added protein is highly advantageous, to ensure proper adherence of the film to the meat after completion of cooking and for the aforementioned  
30 aesthetic purpose of mimicking the glossy appearance imparted by collagen.

The film compositions may also be modified by the addition of compounds for specific purposes, such as

1. anti-microbial;
2. release agents;
- 5 3. colourants (i.e. caramels, oleoresins and other synthetic or natural colourants - iron oxide);
4. flavors and spices.

With particular application to meat processing, black films can be produced using caramel, so that  
10 processors need no longer dip the meat products in a liquid caramel to obtain that colour.

#### Examples of Edible Film Formulations

##### **Example 1**

	<u>Component</u>	<u>Parts</u>
15	Gellan	15
	Locust bean gum	3
	Kappa carrageenan	3
	Konjac	8
	Sodium citrate	3
20	Calcium chloride	2
	Maltodextrin	10
	Glycerol	25
	Water	30

This film exhibited very high tensile strength (140  
25 kg/cm<sup>2</sup>) and good elongation (30%). The film showed outstanding resistance to tear and had excellent handling properties. The film could be removed from a meat product after cooking very easily. (low adhesion)

##### **Example 2**

30	<u>Component</u>	<u>Parts</u>
	Gellan	8
	Kappa carrageenan	10

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	Konjac	3
	Locust bean gum	2
	HVP	7
	High amylose starch	9
5	Sodium citrate	1
	Sorbitan monostearate	1
	Glycerol	25
	Polyethylene glycol	5
	Water	29

10        The film exhibited very high tensile strength (135-140 kg/cm<sup>2</sup>) and elongation (35%), and in particular showed excellent wet tensile strength. Again this film showed outstanding mechanical handling properties and on a meat product had very good adhesion.

15        **Example 3**

	<u>Component</u>	<u>Parts</u>
	Gellan	4
	Kappa carrageenan	7
	Konjac	2
20	Locust bean gum	1
	HVP	7
	High amylose starch	14
	Glycerol	32
	Caramel	4
25	Water	29

30        This film exhibited good tensile strength (110 kg/cm<sup>2</sup>) and elongation (35%) and because of the addition of caramel powder was black in colour. The film had good adhesion properties as well as excellent mechanical handling properties.

**Example 4**

<u>Component</u>	<u>Parts</u>
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	Gellan	10
	Locust bean gum	5
	Kappa carrageenan	8
	Hydrolyzed collagen	10
5	Maltodextrin	7
	Propylene glycol alginate	2
	Silicon dioxide	6
	Sodium citrate	2
	Potassium chloride	2
10	Glycerol	30

This film exhibited moderate tensile strength (100 kg/cm<sup>2</sup>) and good elongation (25%) and utilized collagen to give the film reasonable adhesion to a meat product.

#### Example 5

15	<u>Component</u>	<u>Parts</u>
	Carrageenan	30
	Konjac	15
	Alginate	20
	Glycerol	25
20	Water	10

This film was completely clear and exhibited a very high tensile strength (200 kg/cm<sup>2</sup>) with and elongation of 40%. It showed very high adhesion and could not be removed from the meat product after cooking.

25 Finally, an unexpected advantageous property which we observed in films made according to the invention was efficient heat sealability at 180°C, suggesting usefulness of these films as a replacement for hydroxypropyl methyl cellulose films in casings and food  
30 pouches.

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CLAIMS

1. A liquid composition for casting into an edible film, comprising a uniform mixture of from 2 to 35 wt.% of carrageenan, from 5 to 35 wt.% of a gum selected from the group consisting of Gellan gum and Konjac gum, from 5 to 35 wt.% of a polysaccharide selected from the group consisting of starches and alginates or combinations thereof, from 1 to 35 wt.% of glycerol, and from 8 to 35 wt.% of water.
2. A liquid composition according to claim 1, wherein said polysaccharide comprises a high-amylose starch.
3. A liquid composition according to claim 1, further comprising up to 10 wt.% of locust bean gum.
4. A liquid composition according to any one of claims 1 to 3, further comprising up to 15% of a protein selected from the group consisting of gelatin, soya protein concentrate, soya protein isolate, whey protein concentrate, albumin, vegetable protein, collagen and collagen hydrolysates.
5. A liquid composition according to any one of claims 1 to 4, further comprising up to 25 wt.% of a plasticizing polyhydric alcohol other than glycerol.
6. A liquid composition according to any one of claims 1 to 4, further comprising up to 5 wt.% of sodium citrate.
7. A liquid composition according to any one of claims 1 to 4, further comprising up to 2 wt.% of a salt selected from the group consisting of calcium chloride, potassium chloride and potassium carbonate.

8. An edible casing film prepared by forming and drying a film from a liquid composition according to any one of claims 1 to 7, wherein the proportion of Konjak gum and/or Gellan gum relative to the carrageenan present in said liquid composition has been selected such that the film produced does not disintegrate in water at a temperature in the range of from 70°C to 100°C.
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